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MOSAIC GLASS FROM ST PETER'S, ROME: MANUFACTURING TECHNIQUES AND RAW MATERIALS EMPLOYED IN LATE 16TH-CENTURY ITALIAN OPAQUE GLASS*

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A recent restoration of late 16th-century mosaics in one of the vaults beneath the dome of St Peter's Basilica in Rome allowed sampling and analysis of a group of glass tesserae. The aim of this work is the characterization of opaque coloured glasses possibly produced in Rome. The characteristics of the glass from St Peter's were compared with those of Venetian and Tuscan production, in order to assess possible common origins. Chemical analysis of 30 samples was carried out by electron microprobe, while the nature and morphology of opacifiers were determined by X-ray diffraction and scanning electron microscopy. Almost all the opaque samples were characterized by the presence of SnO_2 crystals. In addition, depending on the colour of the glass, other crystalline phases were identified: lead-tin oxide ($PbSnO_3$) in yellow glass, cuprite (Cu_2O) in orange glass and two calcium-tin silicates with different stoichiometry ($CaSnSiO_5$ and $Ca_3SnSi_2O_9$) in the green-yellow variety. A frame of reference for identifying raw materials and glass-making techniques is provided by textual sources, here examined in comparison with the compositional characteristics of the tesserae from St Peter's.

KEYWORDS: ST PETER'S BASILICA, GLASS, MOSAIC, EMPA, ESEM, XRPD

INTRODUCTION

The resumption of mosaic production in Rome dates back to the pontificate of Pope Gregory XIII (1572–85).

> In the late sixteenth century in Rome the sudden growth of the mosaic technique was the result of a philological recuperation of the origins of Christianity . . . From this point of view, the use of the mosaic medium was borne out by an early Christian figurative tradition, recognized as the bearer of the ideal values of religious purity . . . Thus, independently of the great mosaic art eras of the past, Rome was witness in the modern epoch to a new flowering of the by no means original technique of mosaic production, which has been incorporated in the decorative programme for the Vatican Basilica since its birth. (Cornini 1986)

Pope Gregory XIII, with the decoration of the Cappella Gregoriana (commissioned in 1576), initiated the modern era of mosaics in Rome, which continued with the major project of

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decorating the dome and the vaults of St Peter's Basilica with mosaics (1598–1612) and remaking the paintings of the basilica as mosaics by exactly copying the originals. Besides the 'philological recuperation' of an artistic technique, the aim was also the reproduction of the paintings in materials more resistant to deterioration (Petocchi *et al*. 1981). After the second half of the 17th century Fabio Cristofari began the tradition of making mosaic copies of altarpieces, which persisted into the 18th century. It is important to note that there is a difference between wall mosaics and the techniques used for mosaic altarpieces. The issue of glass translucency and opacity is more relevant in the altarpieces than in the wall mosaics, which are not so closely imitative of paintings.

Since the mosaic tesserae were obtained by a complex and difficult process, the glass was considered a precious material and kept in the store of the Vatican mosaic studio, with meticulous control over incoming and outgoing materials. As regards mosaic glass production, the literature reports a very varied scenario (D'Amelio 2002; Moretti and Salerno 2006; Belmonte and Salerno 2008), and, as can be deduced from the documents, the history of the production of glass tesserae is shaped by the history of the *fornaciari*, definitely proving that glass production in Rome became autonomous in the period between the middle and the end of the 17th century, when the 'workers bound to the Vatican *Fabbrica* acquired the capacity to produce in-house coloured glasses tailored to match the chromatic values of the original paintings' (Cornini 1986). Cornini, from the analyses of just one document, dated 1578, suggests that, initially, coloured glasses were imported from Venice and that skilled mosaicists were recruited from the Venetian Republic, while later the glass was also produced in Rome, exhibiting a higher quality from the perspective of mosaic application. This information led to an over-estimation of the contribution of the Venetian glassmakers (Moretti and Salerno 2006). In fact, together with the unquestionable links with Venice, archive researches indicate that there were already active *ateliers* in Rome in the late 16th century (Belmonte and Salerno 2008). Glass of all colours was certainly produced in Rome at the end of the 17th century, using a different technology and sometimes exhibiting higher qualities and innovations than Venetian glasses (except the ruby-red variety, which continued to be imported from Murano). In general, Roman glasses had higher opacity, while the Venetian ones were translucent. The opacity and homogeneity of colour were of fundamental importance for the Vatican mosaicists, together with the absence of defects such as bubbles, un-melted particles or other irregularities.

Venice also tried to adopt a protectionist policy for its glass-making craft, out of fear of competition from Rome. Although there was a reduction in supply and a rise in price, the ruby-red variety continued to be imported from Venice, although in 1730 one glassmaker in Rome, Alessio Mattioli, succeeded in producing it, albeit at a higher cost. However, relations with Venice should not be exaggerated, and it is very important to note the origin of the glassmakers appointed by the Fabbrica di San Pietro, chosen on the basis of criteria such as the cost and quality of their products. During this period glassmakers arrived from many parts of Italy (from Perugia and other sites in Umbria and Lazio in central Italy, from the village of Cento in north-east Italy, from Noli, near Altare in Liguria, from Vercelli and Cremona in Lombardy, and just one from Venice) to work in Rome, and the resulting glass technology reflects many different traditions (Belmonte and Salerno 2008). Furthermore, each glassmaker had his own furnace and his own trade secrets, making the context quite heterogeneous. There are therefore many reasons to consider the materials employed to produce the mosaics dated to the end of the 16th century as very intriguing, in the period when the Fabbrica di San Pietro was making its initial effort to become a massive, high-quality site for mosaic glass production.

A group of glass tesserae of various colours was collected during a recent restoration of a mosaic dating back to the end of the 16th century (tentatively dated at 1596–98), forming one of the vaults beneath the dome of St Peter's. These tesserae were analysed in order to establish the glass production technique and to compare their composition with contemporaneous mosaic glass from Venice and other sites. Roman mosaic production, which aimed to become independent from Venice, was at this time still in the early stages of a process of technological development that would reach its peak, as has already been stated, only about a century later. Hence, together with the materials of local production, glass imported from Venice could be also present, particularly red tesserae. Moreover, it cannot be excluded that materials markedly different from the originals might have been inserted during restoration interventions in the past. Hence, the aim of this study was also to identify glass of definitely original production and use, differentiating it from other varieties in terms of the production technology used or the era when it was made, before giving a definition of Roman production technology for mosaics.

EXPERIMENTS

Electron microprobe analysis (EMPA)

Chemical analyses were carried out on polished samples using an ARL–SEMQ electron microprobe, equipped with four scanning wavelength spectrometers, sited at the Department of Earth Science of Modena and Reggio Emilia University. The elements analysed were: Si, Ti, Al, Mn, Mg, Fe, Ca, K, Na, P, Cl, S, Sn, Pb, Sb, Cu, Co and Cr. The following geological standards were employed: albite (Na); olivine (Mg); microcline (K, Al); clinopiroxene (Si, Ca); sodalite (Cl), apatite (P); ilmenite (Fe, Ti); spessartine (Mn); cromite (Cr); and cerussite (Pb). Metallic cobalt and metallic antimony were used for Co and Sb calibration, while synthetic cassiterite, $Cu_{94}Sn_6$ alloy and synthetic sulphide $Pb_4Ag_6Sb_6S_{16}$ were used for the calibration of Sn, Cu and S, respectively. The analyses were performed operating at 15 kV, 20 nA, using counting times of 5, 10, 5 s on background–peak–background, respectively. To prevent the known migration phenomenon of alkalis under the electron beam, a 30 μm defocused beam was used. On average, seven points were analysed on each sample to test homogeneity, and the mean value of all the measurements was calculated. The results were processed for matrix effects using the PHI(ρZ) absorption correction of the *Probe* programme (Donovan and Rivers 1990). The measuring accuracy for the analysed elements is better than 3%, while the precision for major constituents is between 1 and 2%, and for the minor constituents it is in the range 2–3%. The results of the chemical analyses expressed in weight% oxides are given in Table 1.

X-ray powder diffraction (XRPD)

The X-ray diffraction experiments were performed on the opaque samples to identify crystalline phases dispersed in the glass matrix. The analyses were carried out with a diffractometer XPERT PRO PanAlytical with Bragg-Brentano geometry θ / θ and CuK_α radiation. The X-ray diffraction experiments were carried out following a non-destructive procedure, directly on the tesserae, using an appropriate multi-purpose sample stage to avoid sampling. The spectra were collected from 5 to 80° 2θ, by using a 0.02 θ step and counting time of 4 s for each step. The results are given in Table 1.

Table 1 *Colour coordinates, X-ray powder diffraction results and chemical analyses in weight % oxides obtained by EMPA for the analysed samples (n.d. = not detected)*

Sample	*Colour*	*L**	*a**	*b**	*Crystalline Phases*	SiO_2	Al_2O_3	TiO_2	*MnO*	*MgO*	*FeO*	*CaO*	Na_2O	K_2O	Sb_2O_3	Cu_2O	*PbO*	SnO_2	*CoO*	SO_3	*Cl*	Cr_2O_3	P_2O_5	*Totals*
SP1	Yellow	75.2	0.7	59.8	SnO_2 $PbSnO_3$	26.86	2.15	0.04	0.17	0.89	0.26	3.61	5.66	1.63	0.03	0.03	53.40	5.98	0.01	n.d.	0.34	0.03	0.12	101.21
SP2	Mustard yellow	69.8	0.3	48.5	SnO_2 $PbSnO_3$	17.11	1.34	0.05	0.06	0.36	0.17	1.53	2.34	0.94	n.d.	0.06	66.32	12.84	0.01	0.03	0.26	0.01	0.04	103.47
SP3	Yellow	67.4	4.7	47.6	SnO_2 $PbSnO_3$ $Pb_9Ca_6[(Si_2O_7)_3(SiO_4)_3]$	23.77	1.84	0.03	0.45	0.63	0.21	1.89	3.41	1.46	0.03	0.04	54.86	12.16	0.01	0.08	0.23	0.01	0.06	101.17
SP4	Yellow	71.7	6.1	55.8	SnO_2 $PbSnO_3$	31.37	2.47	0.09	0.13	0.94	0.24	4.45	5.87	1.98	0.01	0.02	38.26	11.45	0.01	0.01	0.46	n.d.	0.16	97.92
SP5	Orange	45.2	25.5	33.6	SnO_2 Cu_2O	35.43	2.87	0.08	0.08	0.88	0.37	2.95	5.25	3.57	0.15	10.32	34.80	2.64	0.01	0.01	0.54	0.01	0.15	100.11
SP6	Orange	47.4	23.1	32.3	SnO_2 Cu_2O	39.58	3.33	0.09	0.27	1.28	0.89	4.06	6.75	5.59	0.06	4.45	33.56	1.51	0.04	0.12	0.48	0.02	0.29	102.37
SP7	Brick red	32.8	19.7	11.4	Metallic Cu	58.45	4.77	0.25	1.17	2.74	3.04	10.97	13.09	5.12	0.11	1.09	0.18	0.01	n.d.	0.09	0.66	0.04	0.28	102.06
SP8	Brick red	36	23.9	14.4	Metallic Cu	57.76	4.26	0.14	1.52	2.98	3.64	7.66	13.53	6.65	0.04	2.20	1.13	0.43	n.d.	0.26	0.95	0.02	0.30	103.47
SP9	Brown-red	29.9	9.3	3.1	Metallic Cu	61.06	4.05	0.24	1.47	2.84	0.79	10.03	13.86	4.82	0.06	0.84	0.15	0.01	0.01	0.10	0.66	0.01	0.26	101.26
SP10	Orange-grey	39.5	8.8	22.4	–	41.31	3.92	0.17	0.62	1.11	0.65	3.66	4.24	5.67	0.19	4.68	31.71	1.33	n.d.	0.02	0.23	0.02	0.38	99.91
SP11	Orange-green	30.9	1.7	9.3	SnO_2	38.82	2.96	0.08	0.08	0.83	0.36	2.32	5.22	2.65	0.10	6.90	39.52	1.46	n.d.	0.15	0.66	0.01	0.06	102.18
SP12	Green-yellow	33.6	1.7	10.1	SnO_2 $CaSnSiO_5$	52.68	3.84	0.17	1.38	2.38	1.06	7.59	12.07	4.29	0.05	2.51	10.25	2.77	n.d.	0.07	0.61	0.02	0.22	101.96
SP13	Green-yellow	32.6	1.1	7.7	SnO_2 $CaSnSiO_5$	52.00	3.67	0.13	1.48	2.62	1.07	7.22	12.07	5.51	0.01	2.53	10.26	3.55	0.01	0.18	0.70	0.01	0.25	103.27
SP14	Milk-green	59.2	−13	13.4	SnO_2 $Ca_3SnSi_2O_9$	43.34	3.37	0.09	0.06	1.68	0.31	5.36	8.90	3.15	0.12	3.10	26.82	3.98	0.01	0.02	0.91	0.02	0.17	101.41
SP15	Milk-green	48.8	−10.3	9.3	SnO_2	40.02	2.81	0.07	0.03	1.33	0.44	3.22	7.91	3.05	n.d.	6.18	25.29	9.66	n.d.	0.21	1.39	n.d.	0.21	101.82
SP16	Transparent blue	24.2	0.2	−3.7	–	55.24	1.53	0.15	1.27	1.25	0.72	7.65	8.44	10.24	1.88	0.17	6.57	2.47	0.09	0.20	0.56	0.03	1.11	99.57
SP17	Transparent blue	30.2	2.5	−12.1	–	61.30	1.67	0.11	0.58	0.79	0.33	2.41	16.39	7.53	0.07	0.15	4.60	4.19	0.12	0.15	1.16	0.02	0.07	101.64
SP18	Opaque sky blue	37.7	1.9	−17.6	SnO_2	43.92	3.12	0.15	0.82	1.34	1.14	3.98	7.88	3.29	0.01	0.05	21.39	10.24	0.91	0.19	0.68	0.02	0.08	99.21
SP19	Opaque sky blue	48.9	0.1	−16.1	SnO_2	46.89	4.40	0.12	1.73	1.46	0.87	4.83	6.58	5.53	n.d.	0.04	19.79	7.24	0.35	0.16	0.69	0.02	0.17	100.87
SP20	Opaque sky blue	55.1	−2.9	−10.2	SnO_2	30.02	2.51	0.13	0.99	1.28	0.45	3.67	5.72	2.84	n.d.	0.04	20.73	32.42	0.09	0.06	0.49	0.02	0.10	101.56
SP21	Opaque sky blue	61.8	−2.5	−12.7	SnO_2	38.78	2.79	0.12	0.75	1.10	0.43	4.81	4.71	3.21	0.01	0.04	33.40	10.96	0.09	0.09	0.53	0.03	0.12	101.97
SP22	Blue-grey	65.2	−3.7	−4.5	SnO_2	36.96	2.69	0.06	0.81	1.29	0.49	3.25	5.33	3.83	n.d.	0.05	33.47	12.21	0.04	0.19	0.83	0.01	0.15	101.66
SP23	White	86.5	−1.6	7.2	SnO_2	47.19	3.17	0.07	0.34	1.42	0.29	4.51	6.99	4.76	0.00	0.06	17.28	14.07	n.d.	0.12	0.87	0.01	0.10	101.25
SP24	Transparent blue	32.5	−1.7	−6	SnO_2	47.45	2.82	0.14	1.18	1.99	1.54	5.20	9.29	4.55	0.28	0.07	18.60	2.89	0.14	0.04	0.78	0.01	0.12	97.09
SP25	Black	27.7	0.1	−0.7	–	51.47	4.64	0.27	2.15	2.45	1.38	9.33	11.17	6.33	0.08	0.46	7.85	3.19	0.04	0.11	0.55	0.02	0.34	101.83
SP26	Brick red	34.9	15.4	9.7	–	56.54	3.98	0.14	2.07	2.92	5.45	8.05	13.86	6.31	0.06	0.34	0.15	0.02	0.00	0.18	0.88	0.01	0.36	101.32
SP27	Red-black	26.2	1	0.5	$CaSnSiO_5$	51.71	4.23	0.19	0.85	2.26	0.99	7.49	12.15	4.90	0.15	0.51	11.67	3.38	0.04	0.10	0.73	0.00	0.25	101.60
SP28	Transparent blue	24	−0.03	3.1	–	54.10	4.47	0.19	0.97	2.40	0.84	8.11	12.53	5.55	0.11	0.29	7.62	2.92	0.09	0.11	0.77	0.01	0.24	101.32
SP29	Brick red	37	14.6	8	Metallic Cu	52.44	3.98	0.21	1.40	2.45	2.28	7.52	8.07	5.18	0.18	1.21	11.41	2.77	0.03	0.14	0.67	0.01	0.22	100.17
SP30	Intense sky blue	43.1	1.8	−17.5	$Pb_2Sb_2O_7$ $CaSb_2O_6$	60.61	1.36	0.11	0.42	1.24	0.82	6.44	5.69	11.01	8.21	0.02	1.86	0.01	0.19	0.23	0.23	0.02	0.43	98.90

Environmental electron scanning microscopy (ESEM-EDS)

Back-scattered electron images (BSE) and EDS spectra were collected on a low-vacuum ESEM Quanta 200, equipped with an Oxford energy dispersive spectrometer. Owing to the low-vacuum environment, all the analyses were performed directly on the tesserae, thereby avoiding sampling and carbon coating. The analyses were performed using an acceleration voltage of 25 kV and a working distance of 13 mm. The BSE images were mainly collected on opaque glasses to evidence the presence of crystalline opacifying agents in the glass matrix and to define their morphology, and EDS analyses were run to obtain qualitative chemical analysis on these inclusions.

Colour measurement

Owing to the deep opacity of the samples, the colour was measured with a Minolta CM-2600d portable spectrophotometer, to avoid subjective descriptions of the colour. The measurements were performed under diffuse illumination with illuminant D_{65}, illumination/observation geometry d/8°. The colour was expressed in the CIE L*a*b* system. The complete set of coordinates for all the samples is given in Table 1.

RESULTS

Chemical composition

The basic constituents of these glass samples are silica, lead oxide, 'fluxing' alkaline oxides (Na_2O, K_2O) and 'stabilizing' alkaline-earth oxides (CaO, MgO). Alumina is always detectable in non-negligible amounts (from 1.34 to 4.77%); its presence is due to the accessory minerals of the siliceous material (usually feldspars in quartziferous sands), so it can be considered as an accidental component. Iron and manganese oxides are present in levels below 1% in most of the samples (21 and 19 out of 30, respectively). When not deliberately added, the presence of these elements can be considered to be due to impurities in the raw materials. Also the levels of TiO_2, never above 0.3%, are attributable to impurities of heavy minerals.

The main colouring elements, detected in variable concentrations, are copper (responsible for the orange colour), red and green samples, and cobalt (present in blue and light blue tesserae). In some samples Fe and Mn were also intentionally added to obtain the desired shade. In particular, high levels of iron were detected in red samples (SP7, SP8, SP26, SP29: FeO > 2%), in green-yellow samples (SP12 and SP13: FeO ≈ 1%) and in blue samples (SP18, SP24, SP25: FeO ≈ 1.5%). In most of these samples a high level of MnO is also present. Most of the samples contain tin oxide (SnO_2), while antimony oxide is present in appreciable amounts in only two samples (SP16, SP30). The majority of the investigated tesserae are made of lead-rich glass with a highly variable PbO content; only three samples (SP7, SP9 and SP26) contain very low levels of lead (PbO < 0.2%; see Table 1).

In the case of the glass from St Peter's, the flux is mostly sodic, but the potassium content is always significant, as can be observed in the diagram K_2O versus Na_2O (Fig. 1). It can be also seen that in three of the 30 samples potassium is prevalent over sodium. In sodic glass, the ratio Na_2O/K_2O is in the range 1.1 to 3.5, with a mean value of about 2. The ratio between the stabilizers CaO/MgO ranges from 2.4 to 6.1, with a mean value of 3.4, and their relationship is shown in the diagram MgO versus CaO (Fig. 2).

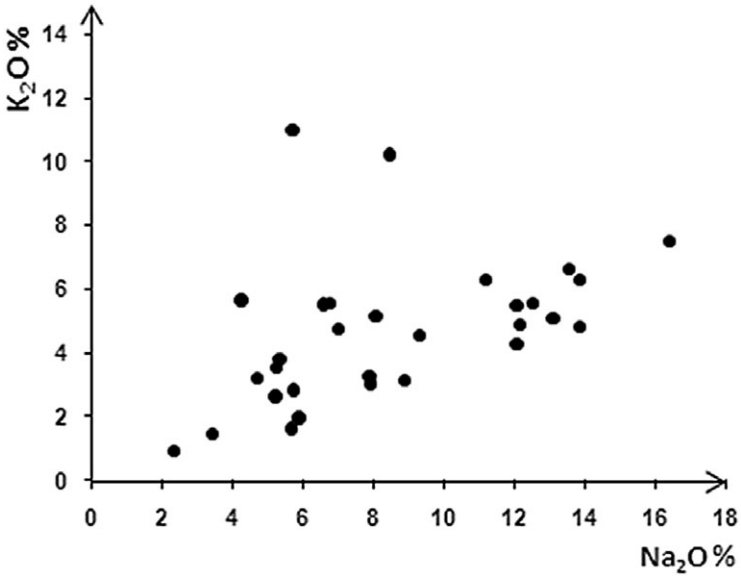

Figure 1 *K_2O versus Na_2O for the analysed samples.*

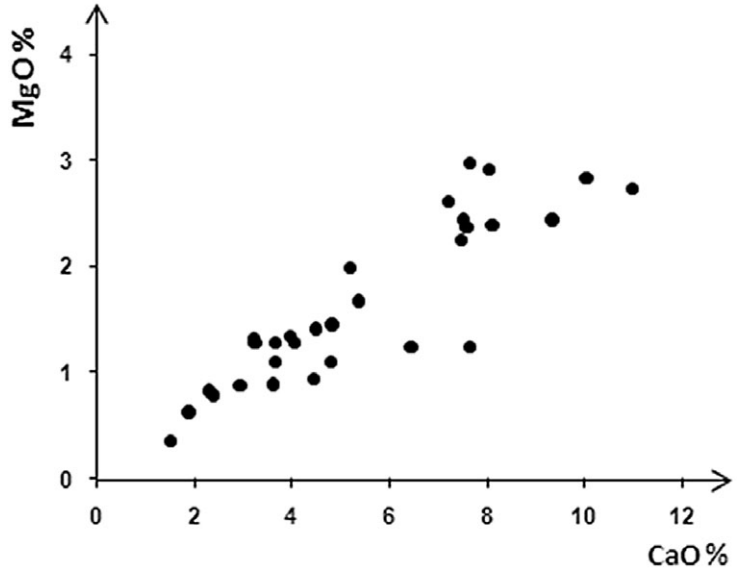

Figure 2 *MgO versus CaO for the analysed samples.*

Opacifiers

The results of the X-ray powder diffraction experiments are given in Table 1. Almost all the tesserae exhibit the presence of dispersed crystalline particles: only the spectra collected on three transparent blue samples (SP16, SP17, SP28), the black tessera SP25, the red SP26 and

the orange-grey SP10 tesserae did not exhibit diffraction peaks. All the yellow tesserae had SnO_2 and $PbSnO_3$ peaks; only tessera SP3 showed the presence of a Pb-Ca silicate ($Pb_9Ca_6[(Si_2O_7)_3(SiO_4)_3]$), probably crystallized drawing calcium and silica from the glass matrix. The BSE images collected on yellow tesserae show the presence of several aggregates whose contrast indicates a very high atomic number; the EDS spectra collected on these particles clearly indicate they are lead stannate (Fig. 3). An intense peak of lead was present also in the spectra collected on the glass matrices. Although the diffraction spectra indicated the presence of cassiterite crystals, it was not possible to distinguish this phase in the BSE image, probably because the crystals were associated with particles of $PbSnO_3$.

SnO_2 was the only crystalline phase detected in several tesserae: orange-green (SP11), milk green (SP15), sky-blue (SP18–21), blue-grey (SP22) and white (SP23). SnO_2 was also present—at a very low level and probably not enough to impart an opaque effect—in the blue transparent tessera SP24. The BSE images reveal that cassiterite crystals were present in some cases as small, rounded aggregates (Fig. 4), and in other cases as elongated needles (SP20; Fig. 5). In some samples SnO_2 was accompanied by other crystalline phases: in green-yellow samples SP12–13 by $CaSnSiO_5$, while in the milk-green sample SP14 by small amounts of $Ca_3SnSi_2O_9$. In the orange samples SP5–6 cassiterite was accompanied by Cu_2O. Almost all the red samples (with the exclusion of SP26 and SP27, which contained no crystalline phases and $CaSnSiO_5$, respectively) contained metallic copper. The BSE images collected on samples SP7–9 and SP29 show the presence of numerous sub-micrometric particles of metallic copper (Fig. 6). From both diffraction spectra and combined SEM–EDS analyses, only sample SP30 showed the presence of Sb-bearing phases, these being $Pb_2Sb_2O_7$ and $CaSb_2O_6$.

Classification on the basis of colours

An initial subdivision of the 30 examined samples can be based on their colour.

Yellow glasses All the yellow samples (SP1, SP2, SP3, SP4)—the colour of sample 2 was defined as 'mustard yellow'—showed remarkable levels of PbO content, in three of the four samples exceeding 50%. They were opacified mainly with cassiterite and $PbSnO_3$ and had relatively low alkali and alkaline-earth contents; the level of the other transition metals was negligible. The ESEM and XRD analyses confirmed the presence of the crystalline phases SnO_2, cassiterite and lead-tin oxide $PbSnO_3$; in one sample a lead-calcium silicate, $Pb_9Ca_6[(Si_2O_7)_3(SiO_4)_3]$, was also found. The colour is evidently due to the yellow phase $PbSnO_3$ (see below).

Orange glasses Samples SP5 and SP6 were orange, with a PbO content of about 33–35% and a remarkable amount of Cu_2O (more than 10% in sample SP5). The level of tin oxide, although quite relevant, was noticeably less than in the previous group. The colour and the opacity were due to the co-presence of Cu_2O (cuprite) and SnO_2 (cassiterite). Samples SP10 and SP11 had orange colour tones, with shades of yellow and green. Their composition was similar to that of the two previous orange glasses, with copper as the colouring element and a low content of tin; no crystalline phases were detected in sample SP10.

Blue glasses Samples SP18, SP19, SP20, SP21, SP22 and SP24 presented a relevant lead oxide content, ranging from 20 to 33%. The amount of SnO_2 was high in almost all these glass samples, with the highest level for sample SP20. Only sample SP24 showed a relatively low level of SnO_2. The diffraction analyses collected on all the samples revealed the presence of cassiterite, respon-

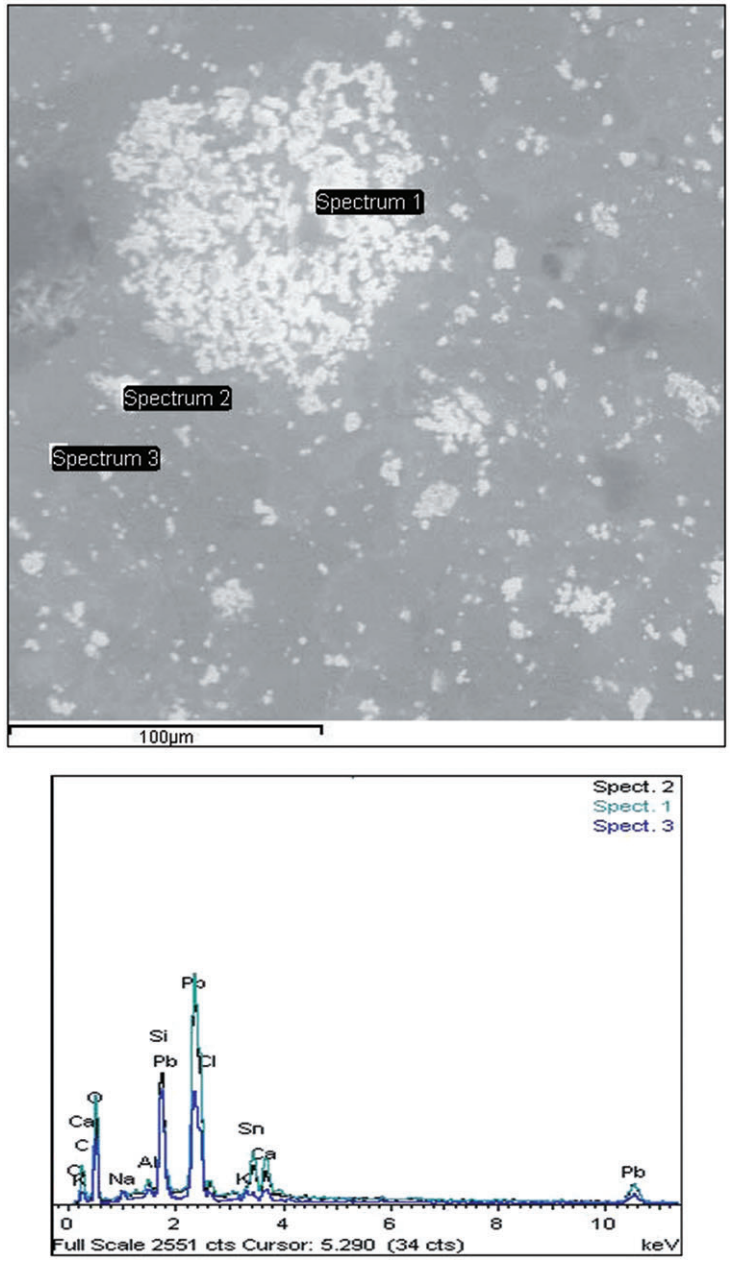

Figure 3 *BSE image and EDS spectra collected on yellow sample SP4.*

sible for the opaque effect. Copper was present only at trace levels, while the contents of iron and manganese oxides were remarkably higher. The blue colour is undoubtedly attributable to the levels of cobalt oxide: considering its high colouring power, it was present in relatively very high levels in tesserae SP18 and SP19 (from 0.35% to 0.91%) and in lower levels (about 0.05 to 0.15%, enough to impart colour) in SP20, SP21, SP22 and SP24.

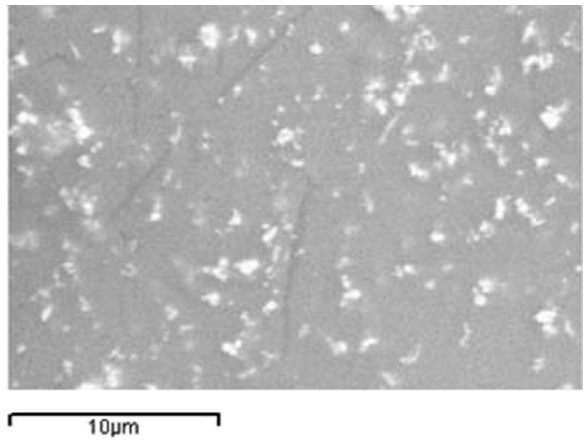

Figure 4 *BSE image collected on white sample SP23.*

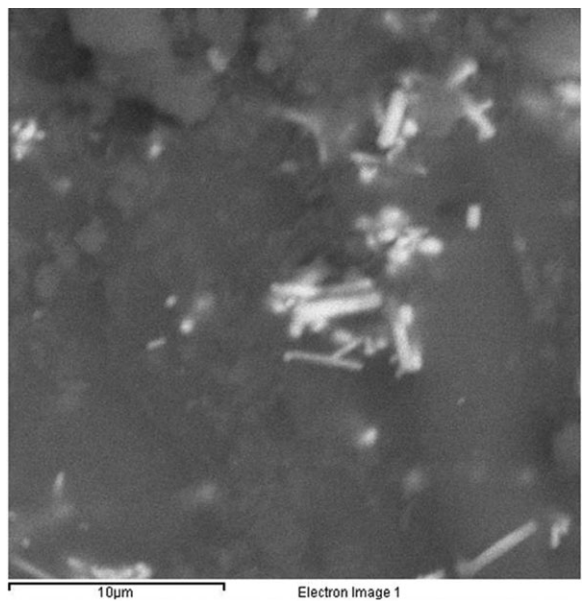

Figure 5 *BSE image collected on sky-blue sample SP20.*

Samples SP28, SP16 and SP17 exhibited a colour defined as 'transparent blue'. They had a PbO content ranging from 4.60% to 7.62%, and SnO_2 from 2.47 to 4.19%. Moreover, they contained low concentrations of iron and copper oxides and the colour was due to cobalt (CoO about 0.1%). Sample SP16, besides having a relatively low amount of lead, had the particular characteristic of containing both tin and antimony, and it belonged to the small group of samples having a potassium oxide content higher than that of sodium oxide.

Sample SP30 also exhibited an intense sky-blue colour, but it presented very peculiar characteristics. In fact, it was the only sample showing a high content of antimony oxide ($Sb_2O_3 > 8\%$),

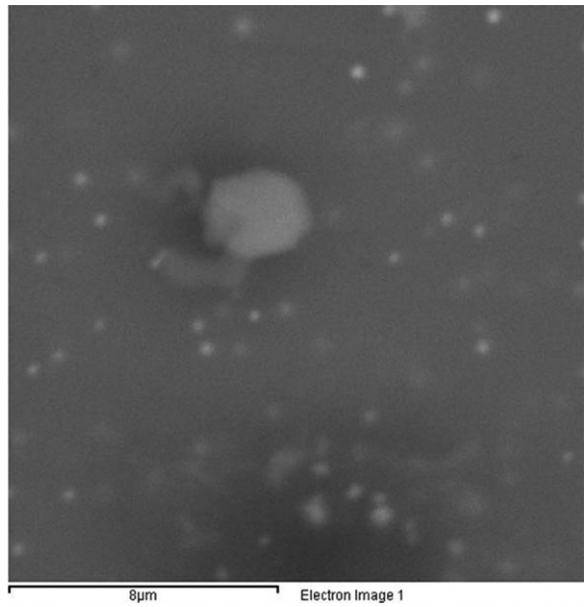

Figure 6 *BSE image collected on red opaque sample SP8.*

consistent with the presence of crystalline $CaSb_2O_6$ and $Pb_2Sb_2O_7$. The latter, even if small in quantity, should give a yellow colour, but this was obscured by the very strong colouring capacity of cobalt, present at a high level in this sample. The potassium oxide level of this sample was very high, double that of sodium oxide.

Green glasses The glass samples SP14 and SP15 had a colour defined as 'milk green', with a PbO content of 25–27% and considerable amounts of tin and copper oxides. Together with the crystalline phase SnO_2, the presence of a tin-calcium silicate ($Ca_3SnSi_2O_9$) was detected in SP14. Samples SP12 and SP13 had a green-yellow colour, a PbO content of about 10% and a fair amount of SnO_2 and Cu_2O; the concentrations of manganese oxide (about 1.5%) and iron oxide (about 1%) were also significant. The crystalline phases were cassiterite and a tin-calcium silicate with stochiometry $CaSnSiO_5$. The significant similarities between these two tesserae suggest that they originated from the same raw glass.

White glass Sample SP23 was the only white tessera; its PbO content was near to 18%. The white opaque effect was certainly due to the almost complete absence of transition elements and to the high concentration of cassiterite crystals dispersed in the matrix (Fig. 4).

Red glass Samples SP7, SP8 and SP9 contained traces of lead (maximum PbO content just above 1%) and exhibited colours defined as 'brick red' (samples SP7 and SP8) and 'brown red' (sample SP9). Tin oxide was very low; the Cu_2O content was over 2% in sample SP8, in which the amount of iron oxide was also relatively high (FeO > 3%), as in sample SP7. The manganese oxide level was fairly high (1–2%) in these samples. The brick-red colour was due to the presence of metallic Cu particles and the relative abundance of iron, probably added as a reducing agent, while in the brown-red glass the amounts of copper and iron were lower and manganese was

very high (MnO about 1.5%). The three glasses were probably obtained under reducing conditions.

Sample SP26 was another brick-red glass, without lead and opacifiers and with a particularly high iron content (FeO > 5%) and a fair amount of manganese (MnO > 2%), while copper was present only as traces. Sample SP29 also had a brick-red colour, but it contained higher levels of PbO (11.41%) and SnO_2 (2.77%) than the other red glass samples. The levels of copper oxide and iron oxide were similar to those found in samples SP7 and SP8; metallic Cu particles, mainly responsible for the colour and the opacity, were revealed by XRD.

Black glass Sample SP25 was a black glass used as a support for gold leaf. The PbO content was about 8% and SnO_2 about 3%; among the colouring elements the manganese (MnO > 2%) and iron (FeO about 1.5%) were relatively abundant; low levels of copper and cobalt were also detectable. The colour of sample SP27 was defined as 'red-black'; this is probably an example of a glass intended to be red but which assumed a blackish tint, an effect well known among glassmakers (see below). SP27 had a higher lead oxide content (PbO > 11%) than the previous sample, and a relatively low amount of manganese oxide (MnO < 1%); $CaSnSiO_5$ was again detected in this glass.

DISCUSSION

The general characteristics of most of the samples examined suggest classification as lead-silicic glass, opacified with tin-bearing crystalline phases and containing very variable amounts of PbO and SnO_2. In the alkaline oxides, potassium oxide was present in non-negligible concentrations, but sodium oxide prevailed in most of the studied samples, indicating the use of plant ashes as flux, generally originating from coastal environments. These characteristics indicate a common basic technology for the production of most of the glass analysed, which can thus be considered original and produced during the period when the mosaic was created. However, there are some samples that showed remarkable differences: first, the samples without lead, in red shades and probably obtained under reducing conditions (presence of metallic Cu particles). Moreover, two glass samples in blue and sky-blue shades had, respectively, a reasonable and a high level of antimony, and a potassium content markedly higher than that of sodium oxide. These samples can be ascribed either to contemporaneous productions at different ateliers, or they may be non-original materials, inserted during restoration work. It is interesting to note the use of antimony in glass of the late 17th century (Verità 2000; Fiori and Vandini 2009) and the production of potassic lead glass in Rome from the mid-17th century (Moretti and Toninato 2001).

On the basis of the criteria outlined above, the technical procedures for obtaining the various colours can be considered, taking into account the fact that in almost all the samples the presence of the crystalline phase cassiterite, SnO_2, was detected. Since PbO and SiO_2 are the most distinctive variables among the sample groups, a correlation between these variables and glass colour was investigated. In Figure 7 the fields of different colours are schematically indicated. The results can be outlined as follows:

- Glass with a high lead content was yellow, as a consequence of the presence of the crystalline phase $PbSnO_3$.
- Orange samples, or those tending towards yellow or green shades, had medium-high lead contents; they contained the crystalline phase cuprite. In addition to these, there were samples with sky-blue or grey-blue colours without copper but quite high in iron and manganese. However, their colours were principally due to the presence of small quantities of cobalt.

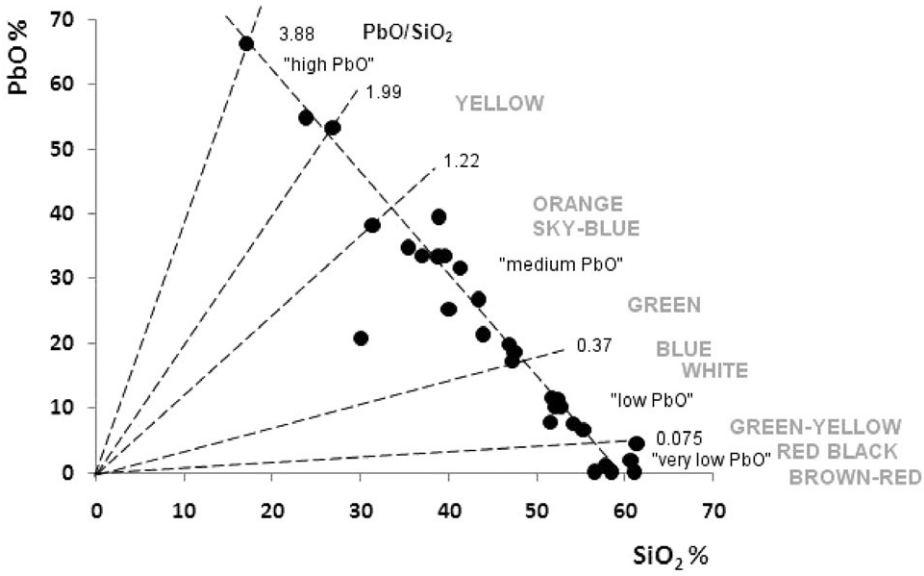

Figure 7 *Schematic representation of the colours as a function of the PbO/SiO_2 ratio.*

• For medium–low lead-content glass, we again found sky-blue or blue glass coloured with cobalt; other glasses of this group were green as a consequence of the presence of copper and iron. A white glass sample contained only traces of colouring elements and was simply opacified with cassiterite.
• The group of tesserae with a low lead content was far more complex: there were green-yellow samples, with a significant presence of copper, iron and manganese, black, with iron and manganese, red-black and brick red, with copper, iron and manganese, and again blue with cobalt.

Tin-based opacifiers

The chemical data, along with the results of XRPD, showed that most of the opaque samples contained Sn-bearing opacifiers. In fact, while antimony-based opacifiers (lead antimonates for yellow, and calcium antimonate for white) had been used from the earliest days of glass production—in the Near East and Egypt around 1500 BC (Mass *et al*. 2002), and through Roman times (Calvi *et al*. 1963; Shortland 2002; Arletti *et al*. 2006a,b)—and, although tin oxide was first introduced as an opacifier as early as the second century BC (Henderson 2000), it was during the fourth century AD that tin-based opacifiers started systematically to replace the antimony-based ones (Tite *et al*. 2007). They spread from the eastern Mediterranean to northern Europe (Turner and Rooksby 1959).

Tin-based opacifiers were also used in Italy from the fifth century AD onwards, but at the same time antimony-based opacifiers continued to be used—or reused—and their disappearance occurred around the 13th century AD (Uboldi and Verità 2003; Fiori *et al*. 2004; Arletti *et al*. 2008). Tin-based opacifiers were used from the fifth to the ninth century AD in the production of yellow and white beads in Anglo-Saxon England (Bayley and Wilthew 1986), Ireland (Henderson 1988) and Germany (Heck and Hoffmann 2000). This kind of opacifier was finally used in the production of Islamic white and yellow enamels in the 12th century (Mason and Tite 1997; Mason 2004) and in the production of Venetian glass in the 13th century (Freestone and Bimson 1995).

In a recent work, Tite and colleagues (2007) examined tin-opacified glass and proposed a reassessment of the method by which these materials may have been produced. The tin-based opacifiers were mostly used for the production of yellow glass as a cubic $PbSnO_3$ phase (yellow) and not in the orthorhombic form Pb_2SnO_4 (white), which does not impart the desired effect. This latter phase (produced by melting lead oxide and tin oxide) is converted into the cubic phase on heating to 850°C in the presence of silica (Rooksby 1964). Interestingly, in an early 15th-century recipe (Kuhn 1968)—comparable to the recipes found for Venetian manufacturing of the 18th and 19th century—a similar two-step procedure was proposed to produce lead-tin yellow opacifiers. The first step consisted in the production of the so-called 'lead-tin calx', by mixing lead metals and tin metals. Then this product was mixed with lead oxide and silica to produce what was referred to as 'lead-tin yellow anime'. This material was crushed and added to a molten colourless glass to produce yellow opaque glass (Tite *et al*. 2007). The 18th- and 19th-century Venetian recipes (Moretti and Hreglich 1984) confirmed that the lead-tin calx was exploited for the production both of yellow anime and of white and blue glass, the latter after the addition of the appropriate colorants. In their paper Tite and colleagues (Tite *et al*. 2007) demonstrate that in a mixture of SiO_2, PbO and SnO_2 the persistence of $PbSnO_3$ is favoured by low PbO/SnO_2 ratios and low levels of silica; otherwise the cubic lead stannate, responsible for the yellow colour, would be transformed into cassiterite at relatively low temperatures (around 750–850°C), preventing the formation of the desired shade. Therefore, for the production of a yellow glass (in which the SiO_2 content is high), it would be essential that the mixing of anime and molten glass occurred at low temperatures and as rapidly as possible, to prevent the dissolution of the lead stannate and the formation of cassiterite.

In our tin-opacified samples the PbO/SnO_2 ratio was very variable and ranged from 0.64 to 27.08 independently of the colour or the opacifier dispersed in the sample. In particular, in our yellow samples the high PbO/SnO_2 ratio (between 3.34 and 8.93) was consistent with the presence of SnO_2—along with $PbSnO_3$—as dispersed crystalline phases, the cassiterite being the result of the initial dissolution of the lead stannate under high temperature. This could suggest that the temperature employed was higher than 750°C. On the contrary, a low PbO/SnO_2 ratio—believed to favour the formation of a lead stannate, preventing the recrystallization of cassiterite—was found in some SnO_2-bearing white (SP23), blue (SP15), light blue (SP20), and green tesserae (SP13). These tesserae were probably produced at higher temperatures and, despite the low PbO/SnO_2 ratio, the formation of cassiterite was facilitated. This is consistent with the co-presence of cassiterite and of the synthetic phase $CaSnSiO_5$ which is formed, in the laboratory, at temperatures as high as 1700 K.

Regarding the other colours, with the exclusion of the red samples, the desired effect was obtained by the presence of SnO_2 crystals—giving the glass an opaque appearance—and the appropriate transition metal (Cu, Fe, Co etc.). The colour of the tesserae SP7, SP8 and SP9 was due to the well-known particles of metallic copper (e.g. Mirti *et al*. 2002; Padovani *et al*. 2003). In these samples the reduction of the copper was probably enhanced by the presence of iron.

GLASS PROVENANCE

Data comparison with contemporaneous tesserae

A further important topic of this study is the definition of the provenance of the glass from the original decoration. In fact, the period of glass manufacture is probably that of the first phase

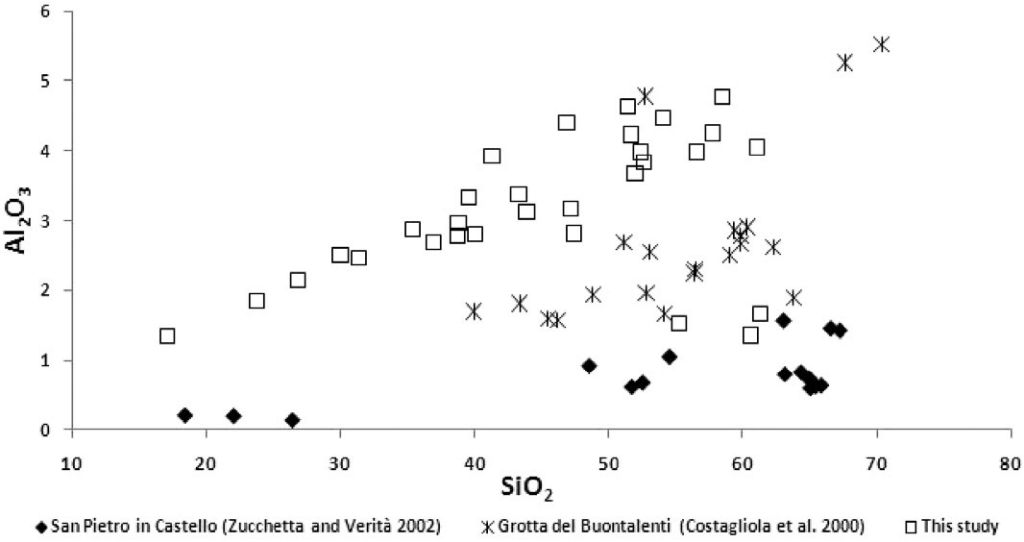

Figure 8 *Al_2O_3 versus SiO_2 for the analysed samples in comparison with those of the mosaic of the basilica of San Pietro in Castello, Venice (Zucchetta and Verità 2002), and the Grotta di Buontalenti, Florence (Costagliola* et al. *2000).*

of production of the mosaics in the St Peter's Basilica, when the glass materials could have been imported from Venice. A recent study concerning the mosaic altarpiece of the Cappella Lando in San Pietro di Castello, Venice (Zucchetta and Verità 2002), reports the chemical characteristics of tesserae definitely produced in Venice. This mosaic, by the mosaicists Francesco and Valerio Zuccato, was finished in 1575 and so pre-dates the St Peter's tesserae examined here by about 20 years. It can thus be taken as a reference point for Venetian production of the period, in order to assess the possibility that the glass used in St Peter's could have come from Venice.

The glass materials of the Venetian altarpiece exhibit quite similar characteristics to those of the Roman glass being studied. They are lead-silicic glass, with a fairly variable PbO content, containing tin-based opacifiers or the same colouring ions for the same chromatic shade. In particular, the constant presence of the crystalline phase SnO_2, the yellow colour due to lead-tin oxide $PbSnO_3$, the presence of cuprite in red-orange samples, the blue shades due to cobalt ions and the green ones due to copper and iron in the glassy phase indicate a similarity with the materials from St Peter's. However, the chemistry of the main components of the glass shows that, especially regarding the K_2O and Al_2O_3 contents, there are remarkable differences between the tesserae from the two decorations. The levels of Al_2O_3 found in the St Peter's glass are very high compared with those from the altarpiece in Venice, which are almost always below 1.5% (Fig. 8). Again, with regard to the alkali contents, there are several points of difference between the two mosaics: the tesserae from St Peter's have higher K_2O and slightly lower Na_2O (Fig. 9). This suggests they were probably produced with a different source of flux.

Another important reference point is provided by a study of mosaic tesserae from the Medici period (late 16th century) from the Grotta del Buontalenti in the Boboli Gardens, Florence (Costagliola *et al*. 2000). In this case, with only a few exceptions—considered by the authors not to belong to this period of production—the glass contains lead in variable quantities and tin-based

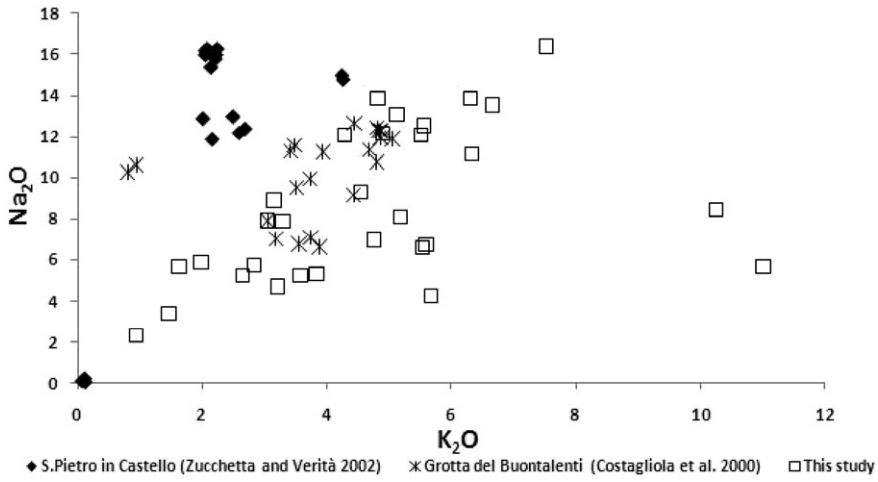

Figure 9 *Na_2O versus K_2O for the analysed samples in comparison with those of the mosaic of the basilica of San Pietro in Castello, Venice (Zucchetta and Verità 2002), and the Grotta di Buontalenti, Florence (Costagliola* et al. *2000).*

opacifiers, as in the tesserae from St Peter's. The chemistry of the fluxing agent matches the data obtained for our samples; in fact, as shown in Figure 9, the levels of Na_2O and of K_2O found for the two decorative projects are very similar—with the exception of two Medici-period samples. However, regarding aluminium, the discrepancy remains. Notwithstanding the fact that the differences are smaller than the Venice decoration, the tesserae from the Grotta del Buontalenti clearly exhibit lower Al_2O_3 than those from Rome.

Although the recipes employed to obtain the desired shades of colour may have been the same—since all the tesserae are silica glass with variable percentages of lead and opacified mainly with Sn-bearing compounds—the differences found in the major components of the glass coming from the three decorative projects do not allow us to consider them as having been produced in a single site, or at least not from a common melt.

Relations with recipes in textual sources

The study of sources from the literature containing recipes for mosaic glass production—with particular reference to lead-rich glasses used for the production of coloured glasses—represents a useful tool for defining the technologies and the materials employed for the St Peter's mosaics. Compared with the common *vitrum blanchum*, the use of lead-rich glass required a finer control of the production processes by glassmakers, especially when the chromophores were added, but the innovation introduced with the lead-rich glass formula provided a more easily workable glass, with a lower melting-point and with a wider colour palette, characterized by greater brilliance.

Starting from the 15th century AD, a tradition of vitreous pastes and enamels can be traced in Venetian glass-making, accompanied by an almost exhaustive production of textual references. There is also evidence of recipe books for the mosaic glass production for St Peter's, but these are definitely of later date (for example the 18th-century Stribal and Pietro Moretti recipe books; see Moretti and Salerno 2006). In Italy, the first mention of lead-rich glass is in the 'Pseudo-Eraclio', from a medieval context; despite various philological problems concerning the text, Chapter 8 of

Book III, dating to the 12th century (Eraclio, ed. C.Garzia Romano, 1996), mentions the mixing procedure for lead in a silicic fusion, albeit in imprecise terms. But the technological transformation of mosaic glass appears to have been achieved only in 1400, in *Dell'arte del vetro per musaico: tre trattatelli dei secoli XIV e XV* (anonymous, edited by G. Milanesi in 1864), in MS 797 of the National State Archive in Florence. Despite the problematic nature of this source—in which many recipes seem wrong and others nonsensical, and the authors are anonymous or only presumed—it represents the first evidence of instructions for the production of lead-rich glass for glazes and enamels, which did not reappear until the recipes of the 16th and 17th centuries. For example, in recipes 9 and 46 of Book II, for the production of *lattimo* (milk glass), the white enamel is opacified with cassiterite (SnO_2) in a lead silicic matrix.

Moving towards more modern times through a series of pointers in the technical treatises of the 16th and 17th centuries, some possible trends can be identified so that, despite inaccuracies and problems of interpretation of the texts, a match can be identified for the analytical data of the tesserae from St Peter's. Almost all the sources for glass recipes from the literature can be ascribed to the Murano tradition of the period: the Renaissance recipe book *Ricettario anonimo* (copy dated to the second half of the 16th century of two recipe books, the former dated to the period between the end of the 15th century and the beginning of the 16th, the latter written before the 15th century; see Moretti and Toninato 2001), the recipe book *Darduin*, dating to about the mid-17th century (see Zecchin 1990), that from Montepellier, dating to 1536 (anonymous, edited by L. Zecchin, 1987) and the recipes of Gasparo Brunoro in the Danzig recipe book of 1645 (see Moretti *et al*. 2004). The exception with respect to the Venetian tradition is the Tuscan Baroque recipe book of the Florentine Antonio Neri, from 1612 (Neri, edited by F. Abbri in 2001).

Chronologically, the earliest evidence of coloured lead-rich glass is again in the *Trattatelli toscani*: in the second volume, recipe 19 ('*A far colore dolce*'), and in the third volume, recipe 8 ('*A mollificare il cristallo*'). In the Darduin (recipe 69) and Montpellier recipe books (recipes 40 and 134) the lead-rich glass is described as the base for a wide chromatic variety of coloured glasses. In the Tuscan source of Antonio Neri there is a detailed description of the difficulties encountered in the production of lead glass varieties (Book IV, recipes 63–64). The same difficulties are also described in the anonymous Venetian recipe book (recipe 13, '*Per fare vetro di piombo che si adopera in molte cose di questo libro*') and, as the title of this recipe suggests, 'To make lead-rich glass that is used for many things in this book', this type of glass is then mentioned many times in the book, including for making mosaic tesserae. In this context it is important to consider the treatise *Trattato de smalti, ò musaico, con li quali sono fatte le figure delle cuppole et altri quadri in San Pietro*, produced between 1694 and 1696 and commissioned from Desiderio de Leli by Carlo Fontana in the early 1690s as part of a broader project, describing the whole range of the decorative scheme for St Peter's. Fontana's editorial project was never completed, but this treatise was found and recently published (see Bonaccorso 2001). It does not contain recipes but gives a few suggestions for the raw materials and procedures for producing mosaic glass.

Very often, common procedures employed in the recipe books are found: analogous prescriptions or simply differently distributed texts indicate that, leaving aside differences in terminology and/or differences in the quantities of the raw materials, the recipes exhibit a common empirical-technological character. With this in mind, and after some general remarks on the raw materials, the different colours of the tesserae from St Peter's analysed in this work are considered below; at the same time an attempt is made to find corroboration of our analysis in the recipes for mosaic glass production.

Basic raw materials

According to most of the recipes, the vitrifying agent mostly took the form of quartziferous pebbles (*cogoli*) from riverbeds (*cogoli del Tesin*—pebbles from the Ticino river—are mentioned in the Venetian recipes); in some cases sands are also cited (*rena del Valdarno*—sand from the Valdarno—is in Neri, Book I, Chapter 8; Biringuccio also reports *renella* in his *Pirotechnia*, edited by C. S. Smith and M. Teach Gnudi). In agreement with Antonio Neri, sand is possibly the vitrifying agent in our samples, given the relatively high content of Al_2O_3, the presence of which is a sign of a less pure quartziferous source. Its mean content in the glasses analysed here was higher than 3%, which is comparable to the data for the glass samples from the Grotta del Buontalenti in Florence (Costagliola *et al*. 2000), but definitely higher if compared with the data of the glasses from San Pietro di Castello in Venice (Zucchetta and Verità 2002).

Similarly, the ratio between Na_2O and K_2O does not match the Venetian glasses, indicating in our case a co-presence of sodic and potassic sources of flux. All the textual sources mention *soda* (not necessarily indicating natural sodium carbonate but possibly sodic plant ashes; see Costagliola *et al*. 2000), sodic ashes or *allume catino*, but we can assume a contribution of potassium introduced in the form of potassium carbonate or wine tartar (potassium bitartrate) (as *grumma di botti, gruma di vino asciutta, sale di tartaro, tartaro calcinato* or grape marc are mentioned in many recipes) and/or from potassic feldspars in the silica source (as observed for some glasses from the Grotta del Buontalenti; see Costagliola *et al*. 2000). Potassic plant ashes are cited in De Leli's treatise (Bonaccorso 2001), in which ashes of fern, broad bean and succulent plants are mentioned together with sodic ashes of *riscolo* (*Salsola kali*). The use of saltpetre was generally excluded, since the evidence of potassic lead glasses is attested in later times (middle and end of the 17th century in Rome and Venice, respectively; see Moretti and Salerno 2006). The sources of stabilizers are not mentioned in the recipes, since they were introduced unwittingly. The sources of lead are generally given as calcined lead litharge (*calcina de piom, piombo brusado*) or metallic lead. In the following discussion more detailed evidence of the correspondence between glass colours and recipes is given, also taking into account the role played by lead.

Colorants and opacifiers

In order to seek a correlation between chemical composition and recipes specifically dedicated to the production of coloured mosaic glasses, similar colours are grouped together, and Table 2 represents a summary of glass colours from St Peter's and the corresponding recipes in textual sources. All the recipes reported in Table 2 were considered in the discussion, but they do not necessarily have a precise correspondence with the composition of the tesserae. Correspondence, or lack of it, is a useful detail when trying to ascribe the production of the different colours to a particular glass-making tradition. For example, the yellow glasses from St Peter's contained extremely high quantities of lead and tin, and the XRPD analyses confirmed the presence of the crystalline phases SnO_2 and $PbSnO_3$. In the recipes the origin of colour and opacity are due to the presence of lead stannate $PbSnO_3$ (see the discussion above on tin-based opacifiers), known as *giallolino* or *zallolin* and obtained by adding calcined lead and tin to the vitrifying mix and subsequent further addition of lead. This practice is mentioned in all the Venetian recipe books but not in the Tuscan *Arte vetraria*, by Neri. The same is true for the orange colour, thoroughly described in the Venetian recipe books but not in Neri. The anonymous text from 1500 the *Ricettario anonimo* (recipe 40) and Brunoro's books are very important textual references since in Europe during the Middle Ages the tradition of producing orange glass was lost and this colour

Table 2 *Samples from St Peter's grouped by colour and recipes found for the corresponding colour: A = Anonymous, 16th century (Moretti and Toninato 2001); B = 'Gasparo Brunoro', 1645 (Moretti* et al. *2004); D = Darduin recipe book, mid-17th century (Zecchin, 1990); M = Montepellier recipe book, 1536 (Anonymous, ed. L. Zecchin, 1987); N = Antonio Neri, Arte vetraria, 1612 (ed. F.Abbri, 2001); T = Dell'arte del vetro per musaico: tre trattatelli dei secoli XIV e XV (G. Milanesi, 1864)*

Colour	*Sample SP (N°)*	*Compositional features*	*Chromophores/opacifiers*	*Corresponding recipes*
Opaque yellow	1, 2, 3, 4	Lead rich glass	/SnO_2, $PbSnO_3$	A18, 39; B157, 160, 278; D73, 79
Opaque orange	5, 6	Lead rich glass	/SnO_2, Cu_2O	A40; B158, 159, D127
Brick red (opaque)	7, 8, 9, 26	Silica glass (lead almost absent)	Fe, Mn/metallic Cu	B153, 173, 174; M110; D129, N58
Brick red	29	Silica glass with lead	Fe, Cu, Mn/ metallic Cu	A34, 42; B58, 61, 200, 298; T73, 74 (book III)
Green yellow	12, 13	Silica glass with lead	Fe, Cu, Mn/$CaSnSiO_5$, SnO_2	A41; B172, 213; M94; N32-35; T76 (Book III)
Orange green, milk -green	10, 11, 14, 15	Lead rich glass	Cu, Mn/ SnO_2	Idem
Transparent blue	16, 17, 24, 28	Various types of glass; sample SP16 with Sb_2O_3. Variable lead content	Co, Fe, Mn/ (SnO_2)	A49; B71, 83, 91, 110; D37, 53; N 49, 50*
Opaque light blue – sky blue	18, 19, 20, 21, 22, 30	Lead rich glass Sample SP30 with Sb_2O_3	Co, Fe, Mn/ SnO_2	A21, 23, 51; B81, 161; D30; N68, 70
Black	25, 27	Silica glass	Mn, Fe, Co/ $CaSnSiO_5$	A43, 44; B211; N51-53
White	23	Silica glass with lead and tin	/SnO_2	A19, 20, 55; B273; D35; M 125, 130; N54, 55; T9, T46 (Book II)

*Note: some of the recipes do not describe lead-bearing glasses, but indicate the same working processes.

was obtained by melting Roman and Byzantine mosaic tesserae. The recipes to obtain an orange colour indicate the addition of a copper colorant (*ramina non brusada*), substantiated in the detected crystalline phase Cu_2O in the group of orange samples, having a matrix very similar to the group of yellow samples but clearly distinguished by the high content of Cu. The recipe books agree on the working technique, also characteristic of the reds: the so-called 'striking', which means a controlled sequence of heating and cooling of the melted glass at the furnace opening (*refogolar*) until cuprite microcrystals are formed, conferring the orange colour in a typical striped appearance.

In order to trace the recipes for the production of the red mosaic tesserae in the textual sources it is convenient to distinguish between the opaque lead-free glass and the transparent lead-rich glass, corresponding to the typologies found in the group of tesserae analysed in this work. The chromophore is metallic copper, detected as a crystalline phase in almost all the samples. The presence of iron is the result of deliberate addition for its reducing action. To obtain a red colour through the addition of copper, glassmakers were forced to have reducing conditions, and some compounds were found appropriate for this purpose (wine tartar, iron oxides/sulphide—*crocum ferri, croco di Marte* and soot). The technology described is generally the same, and is referred to as *spignauro*: the repeated addition to a silica melt of small quantities of the above-mentioned compounds, each time mixing it into the melt. All but one of the tesserae are almost lead-free glasses, belonging to the category known as *sangue di bue* (ox-blood colour), *morello*, *rosso in corpo* or *rosso coppo* (brick red, opaque brown-red glasses). The best description of these lead-free glasses is given in recipe 58 of the Tuscan Neri, in which scrupulous care is requested to avoid the formation of a black tint, specifying the need for reducing conditions (*padellotto vada morto in furnace*). The ingredients are *fritta di cristallo* (frit), *rottami di vetro bianco* (colourless glass scraps) and *calce di stagno* (calcined tin), to which *acciaio calcinato* (calcined steel), *scaglia di ferro* (scales of iron) and *rame calcinato rosso* (calcined red copper) were added. However, an evident discrepancy concerns the addition of tin, not detected in the lead-free red samples from St Peter's. The explanation appears evident when noting that in our sample set the presence of tin was always connected to the presence of lead, indicating the addition of a mixture of these two elements, either as lead-tin calx or *giallolino* or as metallic waste, and, possibly, the unavailability of sources of tin alone. The Venetian tradition of the lead-rich red variety is confirmed by the fact that this transparent lead-rich red glass, traditionally known as *rossicchiero*, is described in many recipes in the Venetian recipe books (although the earliest reference is found in the Book III of *Trattatelli toscani*, recipes 73 and 74). The usual frit of the Murano glassmakers, with the addition of *calcina* of lead and tin, *croco di Marte* with sulphur (see recipe 18 for its preparation), *grippala abbrugiata* (K_2CO_3) and chimney soot described in many recipes corresponds with the composition of the single lead-rich sample from St Peter's: evidence of the difficulties of obtaining this colour, despite the numerous recipes dedicated to this subject.

The green samples from St Peter's exhibit three different shades: orange-green, yellow-green and milk-green, all of the lead-rich type, coloured and opacified with copper, tin and, although less evident, iron. The first textual reference is in the third book of the *Trattatelli toscani*: '*a far verde porro incarnato col vetro*', for lead-rich opaque green tesserae. The recipe describes the preparation of the colorant *ramino* from the flakes produced by copper beating on an anvil. The other recipe books offer many analogous recipes, mostly evident in the anonymous book and Brunoro's books, in which the prescriptions are relative to mosaic glass: the lead-rich glass base is always mentioned and the colorants are *ramina brusada* or *ramina di tre cotte* (CuO) and *giallolino*, which could be added also in the form of yellow glass scraps. This last ingredient is the most meaningful for the comparison with the composition of the tesserae from St Peter's, in

which the opacifiers detected by XRPD were tin-based. For the two samples of a lactescent green tint, the addition of powdered *lattimo* is not to be excluded, a white lead-rich glass being described in the same recipe books as a base for all the colours. In Book I of Neri there is a description of the production of green glasses, either with or without lead. In particular, recipe 35 shows some similarities with the other Tuscan source of the *Trattatelli toscani* (Book III, recipe 76), confirming the attention of the Tuscan tradition to the production of these coloured glasses.

The group of light blue or azure tesserae was characterized by high contents of lead and tin (a clear exception is the sample SP30, made from an antimony-rich potassic glass); the main chromophore was cobalt, and the opacifier was SnO_2. The textual sources provide abundant recipes to obtain this colour, either for enamels for jewellery, or opaque mosaic glasses or imitations of semi-precious stones. The Venetian recipe books prescribe a base of *lattimo*, to which *zaffera* (roasted cobaltite, CoO), sometimes with copper oxide, was added. Since copper was not detectable at appreciable levels in the light blue samples from St Peter's, the most interesting comparison is found in the Darduin recipe book, in which indications are given for the *Turchino in corpo*: frit plus lead-tin calx and *zaffera* are mixed without mentioning copper (*ramina*), in close agreement with the analytical data from St Peter's. Antonio Neri, in Book IV of his work, dedicated to lead-rich glasses, indicates the use of *gazzera*, a copper oxide with the addition of *zaffera*; in this case the Tuscan textual source, although considering the light blue colour, is not in agreement with the chemical data for the discrepancy regarding copper. Neri's recipes instead present a very good point of reference for the blue glasses from St Peter's (samples predominantly made from a lead-rich sodic glass, using tin with cobalt as the main colorant): in recipe 70 he describes *colore di zaffiro, in vetro di piombo* (sapphire colour in lead-rich glass), in which lead oxide, *zaffera* and *denaro* (denarius of manganese) are added to a silicic frit. As with the red glasses, the disagreement relates to SnO_2, in this case not mentioned in the recipe but present in the chemical composition.

Again, Neri's volume, from Tuscany, gives very good indications for the two black glasses found in St Peter's, the colour of which was due to the presence of different chromophores: mainly manganese and iron and also a non-negligible content of cobalt. Neri presents three interesting recipes (recipes 51 to 53). In the first the colorants are manganese and cobalt, to be added to scraps of variably coloured glass; recipe 52 is the only one that prescribes lead and tin *calcina*, together with *acciaio in polvere* (steel powder) and *scaglia di ferro* (scales of iron) as chromophores; in recipe 53 the colorants for the black glass are manganese and *greppola di vino rosso* (red wine tartar). Therefore, the combination of the three above-mentioned recipes represents the best point of comparison for the composition of the black mosaic glasses from St Peter's. The Venetian tradition also offers numerous textual references for black glass production, giving the same instructions for the chromophores, but with significant differences as regards the lead-free base glass and the addition of calcined bones, which were already excluded in the samples of the present study, owing to the low content of P_2O_5.

The final case regards the white sample analysed in the group of tesserae from St Peter's. De Leli's treatise considers white glass (obtained by adding lead and tin to a base glass) as the base of all the other colours. The recipe books do not expressly mention white glass and instead use the term *lattimo*, a white enamel prepared with calcined lead and tin, employed as a base glass for the preparation of jewellery enamels and for many opaque mosaic glasses. The oldest references are found in 14th-century Venetian documents regarding payments (Moretti *et al*. 2004), while the first recipes appear in the *Trattatelli toscani*. The Venetian and Tuscan sources agree in the description of the raw materials and production technologies, indicating a mix of lead-tin calx and common glass. The proportions indicated in the various recipes are quite contradictory, and none of them corresponds to the case being studied here. Despite the variabil-

ity of the proportions, it is nevertheless clear that the white glass from St Peter's fits perfectly into the 15th- to 17th-century lead-tin opaque glass tradition described in recipes.

In conclusion, by comparing the textual sources with the chemical compositions, it is not possible to identify one particular set of recipes that shows a closer overall match than the others. One of the main difficulties is, of course, the variability in the number, care and level of detail of the descriptions in the different recipe books, but some observations are worthwhile. It is clear that the yellow, orange and lead-rich red glasses belong to the Venetian tradition, since their preparation is not discussed in the Tuscan sources. However, there are differences in some ingredients, mostly regarding the addition of calcined bones, which rule out the immediate assumption that these glasses are of Venetian manufacture. On the other hand, some of the instructions given by the Tuscan author Neri to obtain other colours are in very close agreement with the chemical composition of the glasses from St Peter's, if not in every case (the question of discrepancies in the addition of tin in red and blue glasses is an obvious example). This evidence, and the indication of sand as a vitrifying raw material in Neri's text, support the idea of a link between St Peter's and the Tuscan tradition.

CONCLUSIONS

As indicated in archive documents, from the late 16th century (1578), there were active glass furnaces in Rome. Over time, glassmakers arrived from different areas of Italy, each bringing their own tradition. Although there is continuing evidence of links with the Venetian tradition for at least two centuries, it is simplistic to assume that the process was limited to the importing of glass (or glass technology) from Venice. At the present stage of research, establishing a precise provenance for the glass-making tradition appears an over-ambitious goal. The chemical data obtained and their comparison with samples of contemporaneous 'Venetian' mosaic glass do not support the hypothesis of importation of glass from Venice, or of Tuscan production. However, while the chemistry of the mosaics from St Peter's is different from both Venetian and Tuscan contemporaneous glass, a comparison of the chemical data with sources in the literature reporting Venetian and Tuscan glass recipes seems to suggest compatibility with the use of these recipes for the St Peter's tesserae, although closer inspection reveals many contaminations and no precise correspondence. The data obtained in the present study seem to suggest the existence of a dedicated production, which on the basis of the information currently available cannot be geographically located. However, the studied data, the considerable extension of the time of mosaic glass production and the extraordinary importance of the site could easily lead to the conclusion that the glass-making production of the Fabbrica di San Pietro was an independent and autonomous tradition.

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